

Electronic Version

Stylesheet Version v1.1.1



## Description

### **[Colorant Removal from Polymeric Fibers]**

#### **BACKGROUND OF INVENTION**

[0001] Articles produced from virgin polymers are typically colored for both practical and aesthetic reasons. Articles other than fibers typically have colorants dispersed throughout the article. In addition, polyolefin fibers have colorants dispersed throughout the fiber because surface dyeing techniques have proven unsuccessful. Polyester, Nylon 6, and Nylon 6,6 fibers are typically colored by dyeing the surface of the already-formed fibers.

[0002] One significant limitation to the utility of recycled polymer derived from post-consumer fibers is the color difference between virgin polymer and recycled polymer. The dyes on the surface of recycled polymeric fibers may significantly decrease the suitability of the constituent polymer for reuse because color is not necessarily removed during conventional recycling processes.

[0003] A process directed to the removal of surface colorants is applicable only to certain polymeric fibers. However, a

substantial quantity of polyamide fiber from post consumer carpet is potentially available for recycle. Approximately 40% of the face fibers in post residential carpet waste in the United States is surface dyed Nylon 6, while another 40% is surface dyed Nylon 6,6.

[0004] Processes for stripping dyes from fabric include U.S. 4,227,881 (Fono) which discloses a process for stripping dyes from textile fabric which involving heating an aqueous solution of an ammonium salt, a sulfite salt and an organic sulfonate, such as sodium hydroxymethane sulfonate, to at least 60 degrees Celsius and adding the dyed fabric to the heated solution while maintaining the temperature of the solution. This process is believed to result in less than satisfactory colorant removal.

[0005] U.S. Pat. No. 4,783,193 (Pensa) teaches a process for stripping color from synthetic polymer products by contacting the colored polymer with a chemical system including unstable dispersions of alkyl halides and aqueous solutions of bleaching/oxidizing agents to which specified quantities of acids and surfactant/wetting agents are added. The use of this chemical system may restrict the recyclability of the decolorized polymeric materials.

[0006] U.S. Pat. No. 5,989,296 (Patton) teaches a process for removing indigo dye from denim scrap by extracting the fabric with an

organic solvent such as 1,1,2-trichloroethane in which the indigo dye is soluble at elevated temperatures, the solvent is cooled and extracted with an aqueous phase containing a reducing agent, and the aqueous phase is treated to oxidize and recover the indigo dye. This process is applicable only to indigo dyes.

[0007] U.S. Patent No. 6,083,283 (Berkstresser, IV) teaches a process for removing color and extracting dyes from polymeric materials by contacting them with a swelling agent under conditions such that the swelling agent interrupts the molecular forces within the polymer matrix and opens the polymer structure sufficiently to remove natural and synthetic pigments dispersed throughout a polymeric article, thus it has wide applicability for colored polymeric articles other than surface dyed fibers. The use of swelling agents which penetrate throughout the polymer matrix to remove surface dyes is potentially undesirable because completely removing them after decolorization of the fiber would be expected to involve extensive washing.

[0008] Thus an unmet need exists for a cost-effective and environmentally friendly process for removing surface colorant from synthetic polymeric fibers without degrading the fiber, or otherwise compromising the polymeric material's suitability for recycling and re-use. This unmet need exists particularly for a

process to remove surface colorants from the substantial surface dyed Nylon 6 and Nylon 6,6 fiber component of post-residential waste carpet. A process that can be conducted at atmospheric pressure is most attractive.

## **SUMMARY OF INVENTION**

[0009] The present invention is directed to a method for removing surface dyes and colorants from polymeric materials. More particularly, the present invention is directed to a method for decolorizing Nylon 6, and Nylon 6,6 fibers for the purpose of increasing the suitability of the constituent polymers for subsequent reuse.

[0010] The present invention is directed to a cost-effective and environmentally friendly process for removing surface stains and dye-imparted color from colored polymeric fibers.

[0011] The process and composition of the present invention are particularly useful in the recycling of thermoplastic materials. Materials decolorized by the process of the present invention may be utilized in place of or blended with virgin thermoplastics in any known thermoplastics applications including extruding the melted material to form fiber which may be dyed.

[0012] It was unexpectedly discovered that a dye or other colorant can be substantially removed from the surface of polymeric

materials, particularly fibers, by contacting the materials with an organic ester solvent stripping composition containing a cyclic ester, particularly ethylene carbonate, propylene carbonate, or butylene carbonate, at a temperature below the boiling temperature of the ester solvent stripping composition to effect the release of dye or other colorant from the surface of the polymer. Thus the process can be carried out at ambient pressure.

[0013] Copending patent application 10/708,479 (Mauldin) discloses that polyester polymer is decomposed when heated in the presence of a cyclic ester such as propylene carbonate to form an admixture having utility as an industrial solvent. This novel solvent composition has been found to remove colorants from the surface of polyamide polymer fibers. In this embodiment of the invention, the colored polyamide fibers are contacted with the ester solvent stripping composition at a temperature between about 90 degrees Celsius and about 220 degrees Celsius. Optionally, the decolored polymeric material can be subsequently washed with a polar cyclic ester such as ethylene carbonate, propylene carbonate, butylene carbonate, and mixtures thereof, containing no dissolved colorant.

[0014] The process of the present invention includes contacting colored polyamide fibers with at least one ester solvent stripping composition under conditions sufficient to effect

release of the colorant from the surface of the fibers. The process is especially useful for quickly and thoroughly removing colorants from surface dyed polyamide fibers as part of a recycling process for such fibers. Colorants are considered to be any dye, pigment or colored composition or combinations thereof that may intentionally or accidentally color or stain polymeric materials, while dyes are considered to be organic materials which impart color to a polymer and which chemically bond to the polymer surface primarily by ionic mechanisms. The process of the present invention removes colorants from the surface of polyamide fibers without substantially degrading Nylon 6 or Nylon 6,6 polyamide polymers, thus allowing for their recovery and reuse.

[0015]

In one embodiment of the present invention, a process for recycling colored polyamide fibers comprises the steps of shearing or cutting Nylon 6 or Nylon 6,6 face fibers from post consumer carpet waste, then contacting the colored polyamide fibers with an ester stripping solvent solution at a temperature effective to remove colorant from the surface of the polymeric material. In another embodiment, a process for recycling colored polyamide fibers comprises the steps of shredding the entire carpet and grinding the face fibers and backing components to yield individual fibers commingled with discrete particles of backing materials, then separating the fibers from

the non-fibrous components before contacting the colored polyamide fibers with an ester stripping solvent solution at a temperature effective to remove colorant from the surface of the polymeric material. These two embodiments are especially useful in recycling the surface dyed polyamide face fiber component of post-consumer carpet.

[0016] Another embodiment of the process of the present invention which can allow recycling of the ester solvent stripping composition used to decolorize colored polyamide material comprising: (a) removal of colorant from colored polyamide fibers utilizing an ester solvent stripping composition further containing an alcohol that separates as an immiscible liquid phase as the ester solvent stripping composition is cooled to ambient temperature; (b) cooling the ester solvent stripping composition to a temperature between about 20 degrees and about 90 degrees Celsius, whereby the solution separates into an ester phase and an alcohol phase containing colorant; and (c) removing colorant from the alcohol phase through further separate processing while the ester phase is immediately available for reuse as a component of the ester solvent stripping composition.

[0017] In practicing the present invention to decolorize the colored polymeric face fiber component of post consumer carpet waste, the process of decolorization should preferably be

preceded by one or more of the preliminary steps of (a) physically segregating carpet pieces having Nylon 6, or Nylon 6,6 face fibers; (b) cleaning the group of carpet pieces containing only one of Nylon 6 or Nylon 6,6 face fibers from step (a) by mechanically separating dirt and other loosely-attached foreign materials; (c) separating the Nylon 6 or Nylon 6,6 face fibers from the backing of the carpet by a method selected from the group consisting of mechanical shearing, melt-cutting with a hot wire, melt-cutting with a laser, shredding followed by grinding and air elutriation, and combinations thereof; and (d) cutting, shearing, or grinding the colored fibers into fibrous particles having reduced size.

[0018] The present invention has a number of advantages over prior art decolorization methods.

[0019] The present invention does not substantially degrade the polymer and therefore recovered polymer can be used in new polymeric materials or articles in place of virgin polymer. The stripping solvents of the present invention laden with colorants can be thoroughly removed from the surface of the fibers with relative ease because the stripping agent does not penetrate into the fibers and disrupt the molecular forces within the polymer matrix sufficiently to result in an opening of the polymer structure.

## DETAILED DESCRIPTION

[0020] The process of the present invention includes contacting colored polymeric fibers with at least one ester solvent stripping composition under conditions so as to effect the release of a dye or other colorant from the surface of the polymeric material. The amount of the ester solvent stripping composition and the conditions under which the contacting takes place are selected so that the polymeric material does not undergo substantial destruction or degradation. The contacting step is most preferably performed at ambient pressure.

[0021] As will be apparent to one skilled in the art, the combined effect of temperature and the formulation of a suitable contacting composition can be used to control the processes of the present invention. Thus variation and optimization of the contacting composition, and the temperature, time, and repetition conditions of the contacting process in order to maximize the decolorizing effect of the contacting composition are considered to be within the scope of the present invention. It should be noted that since the process is conducted at ambient pressure, only ester solvent stripping compositions containing esters and alcohols that boil at relatively high temperatures can be used.

[0022] A preferred practice of this invention utilizes the temperature dependence of the solubility of dyes in the ester solvent

stripping compositions to effect separation of dyes from the ester solvent stripping compositions. The dyes are removed as particulate precipitates, thus allowing recycling of the ester solvent stripping compositions.

[0023]

Alternatively, the dissolved dyes can be removed from the ester solvent stripping compositions by prior art techniques such as adsorption onto activated carbon or some other solid surface, chemical destruction, or electrolytic coagulation. The residence time for contacting the colored polymeric material with the contacting composition during the contacting step may be controlled to ensure the desired degree of color removal.

Suitable residence times for the contacting step will depend upon the conditions of the contacting step. The preferred residence time is at least about 1/2 minute and no greater than about 20 minutes, more preferably about 3 to 10 minutes. The contacting step in these embodiments may include a plurality of contacting stages wherein the colored polymeric material is contacted with an ester solvent stripping composition at each stage. One skilled in the art would appreciate that the residence time varies depending on the temperature and other conditions in order to achieve the results of the present invention. The process of the present invention may further include a washing step, wherein any residual dye, colorant, or ester solvent stripping composition is removed. Suitable

washing agents should at least partially solubilize residual dye, colorant or ester solvent stripping composition without harming the decolorized polymeric material. Washing agents should be polar liquids and preferably are selected from the group including ethylene carbonate and propylene carbonate. Water, C.<sub>sub.1</sub> to C.<sub>sub.4</sub> aliphatic alcohols, and mixtures thereof may also be used. An after treatment wash with an aqueous 0.1% to 0.3% sodium hydrosulfite solution may also be employed to enhance final polymer color. The contacting step may be performed using a variety of techniques that will be apparent to one of ordinary skill in the art. Such techniques include immersing the colored polymeric material in the ester solvent stripping composition, spraying an effective amount of ester solvent stripping composition onto the polymeric material, and other similar such techniques. Further the contacting step may be carried out in lots in a batch-wise manner or it may be carried out in a continuous manner. In an especially preferred embodiment, dyed nylon fiber is contacted with the solvent composition disclosed in copending patent application 10/708,479 (Mauldin) at a temperature of at least about 130 degrees Celsius for a period of about 0.5 to 5 minutes. A series of two or three sequential treatments can be employed to improve the final polymer product color.

[0024]

The following examples are included to demonstrate preferred

embodiments of the invention. Those skilled in the art should, in light of the present disclosure, appreciate that many changes can be made in the specific embodiments which are disclosed and still obtain a like or similar result without departing from the spirit and scope of the invention.

[0025] EXAMPLE 1 A deep red surface-dyed Nylon 6 yarn was cut into about 1 inch lengths and 10 grams of the yarn was placed into an Erlenmeyer flask with 100 grams of solvent prepared by admixing 100 grams of Propylene Carbonate with 20 grams of Poly(ethylene terephthalate) yarn and heating the admixture to 230 degrees Celsius for 15 minutes. The Nylon 6 yarn pieces were immersed in the ester solvent composition, heated to a temperature of 160 degrees Celsius, whereupon the solvent was separated from the Nylon 6 fibers by filtration. The Nylon 6 yarn pieces were visibly lighter in color and the ester solvent composition was observed have a strong red color.

[0026] EXAMPLE 2 Colored Nylon 6,6 carpet fibers recovered from post-residential carpet waste were obtained from a commercial broker of recycled thermoplastic materials. Ten grams of these fibers were selected to obtain fibers of at least four distinct colors including red, blue, beige, and brown; the fibers were placed into an Erlenmeyer flask with 100 grams of solvent composed of 70 grams of Propylene Carbonate and 30 grams of "Soygold 1000" methyl ester of soybean oil. The fibers were

immersed in the ester solvent and heated to a temperature of 200 degrees Celsius, whereupon the solvent was separated from the Nylon 6,6 fibers by filtration. The fibers had assumed a uniform light grey appearance while the ester solvent composition was observed to have a brown coloration.

[0027]

EXAMPLE 3 Surface-dyed Nylon 6 carpet fibers recovered from industrial commercial carpet waste were obtained from a commercial carpet manufacturer. The fibers were deep blue in color. Ten grams of these fibers were placed into an Erlenmeyer flask with 100 grams of solvent composed of 80 grams of Propylene Carbonate and 20 grams of 2-Octanol. The fibers were immersed in the solvent and heated to a temperature of 130 degrees Celsius, whereupon the solvent was separated from the Nylon 6 fibers by filtration. The fibers had assumed an off-white color. The solvent was observed to have a blue color. Upon cooling below 55 degrees Celsius, the solvent separated into 2 liquid phases with the greatest volume represented by the lower phase. The 2-Octanol phase was substantially darker blue in color than the Propylene Carbonate phase. While the compositions and methods of this invention have been described in terms of preferred embodiments, the present invention may be embodied in other specific forms without departing from the spirit or essential attributes thereof, and accordingly, reference should be made to the

appended claims, rather than to the foregoing specification, as indicating the scope of the invention.